

PATENT SPECIFICATION

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(54) USE OF SYNTHETIC CLAY CONTAINING NO LITHIUM AS SOIL ANTI-REDEPOSITION AGENT, IN DETERGENTS

(71) We, PFIZER INC., a Corporation organized under the laws of the State of Delaware, United States of America, of 235 East 42nd Street, New York 17, State of New York, United States of America, do hereby declare the invention, for which we pray that a Patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

Modern day synthetic detergents are usually very effective in loosening soil particles in dirty clothing fabrics and removing the soil into the wash water during laundering operations, the soil being suspended in the aqueous detergent solution. However, some of this soil will be redeposited on the fabric during the laundering, causing a loss of whiteness in the fabric and a grey appearance.

One of the more common soil anti-redeposition (SARD) agents used commercially today in detergent formulations is carboxymethylcellulose (CMC) or its alkali metal salt, usually being sodium. Another common SARD agent used in commercial detergent formulations is polyvinylpyrrolidone (PVP). However, both SARD agents are relatively expensive, and in addition, do not seem to be uniformly effective against all types of fabrics; CMC not being uniformly effective on fabrics having synthetic fibers, and PVP not being totally effective on cotton fabrics.

In contrast to the prior art, it has been surprisingly found that SARD properties can be imparted to a detergent formulation, whereby the redeposition of soil upon a fabric from the aqueous washing solution of said formulation is substantially reduced, by incorporating in the formulation an effective amount of a compound having the formula

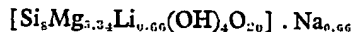


wherein X is from about 0.5 to 0.9. Prefer-

ably the compound is used in the formulation in a form wherein about 90% of the particles of the compound have a particle size smaller than about 325 mesh. More preferably, X is 0.7 in the general formula above and this compound is particularly effective on cotton fabrics and synthetic fabrics.

Most clay minerals, as found naturally, are in an impure state and the complete purification of some is difficult and expensive and, in some cases, impossible. Further, there are occasions on which the supply of a clay mineral of a particular chemical composition, either pure or impure, is insufficient. Thus, it is desirable to be able to manufacture synthetic clay-like minerals in a substantially pure form and of pure white color.

It is of particular interest to be able to manufacture synthetic clay-like minerals having rheological properties similar to or better than those of hectorite, as natural hectorite has valuable properties but large quantities of hectorite are not available. In any event natural hectorite is mixed with impurities the removal of some at least of which is extremely difficult. The naturally-occurring clay, hectorite, has the formula



wherein F may replace some OH. The clay-like materials used in the present invention have structural formulas very similar to natural hectorite, X in the general formula being 0.66, and H replacing Li in the formula for hectorite.

Two methods are known for synthesizing hectorite-type clay minerals. One is described in Granquist and Pollack, "Clays and Clay Minerals", National Academy of Science, National Research Council Publication, vol. 8, pp. 150—69 (1960). The other is described by Strese and Hofmann in *Z. Anorg. Chem.*, 247, pp. 65—95 (1941).

The clay-like materials of the present inven-

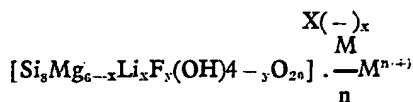
tion may be advantageously prepared by means of a process which comprises:

- (a) forming an aqueous slurry from
 - i. a water-soluble magnesium salt,
 - ii. sodium silicate,
 - iii. sodium carbonate or sodium hydroxide
 and the aqueous slurry being formed by co-precipitation by slowly combining the said magnesium salt and the said sodium silicate and the said sodium carbonate or sodium hydroxide, with heating and agitation (e.g. 200°F.), the slurry containing stoichiometric amounts of all the cations and anions that are desired to be present in the final product;
 - (b) taking the aqueous slurry so formed (e.g. after about 2—4 hours), washing it free from soluble salts, filtering, and adding aqueous NaOH to maintain an alkaline solution;
 - (c) Autoclaving the slurry (e.g. at a pressure about 100—200 psig and a corresponding steam temperature about 338—388°F.) for about 10—20 hours to crystallize the synthetic mineral-like clay; and
 - (d) drying and grinding the finished product.
- Alternatively, step (a) can be performed in a pebble mill without heating in which the slurry is formed by blending for about 1 hour and then processed according to steps (b), (c), and (d) above.

Many variations of the above process can be made by anyone skilled in the art.

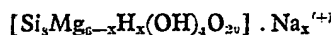
- Any suitable magnesium salt may be used for introducing the Mg cation into the solution such as magnesium chloride, magnesium sulfate, and magnesium nitrate. It is preferred to use $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$. The silicon cation may be introduced in the form of any suitable silicate. The sodium cation is introduced preferably in the form of Na_2CO_3 , NaOH , or Na_2O .

- The process for preparing the clay-like minerals of the invention has broad applicability in the preparation of compounds which are similar in structure to other naturally occurring clays (e.g. other montmorillonites). Thus by introducing the correct stoichiometric amounts of cations and anions in the initial slurry, it may be possible to prepare compounds having the general formula



where M can be any cation having a valence, n, (e.g. K, Cu, Ca, Co, Al, Li, etc.).

- The synthetic clay-like materials having the general formula



where X is from about 0.5 to 0.9 are preferred

for use as soil anti-redeposition (SARD) agent in detergent formulations. Most preferably, not less than about 90% of the SARD agent particles in the detergent formulation have a particle size smaller than 325 mesh. Furthermore, it has been surprisingly found that when X is 0.7, this particular compound is more effective on cotton and synthetics than the commercial SARD agent, CMC, especially at the small particle size distribution mentioned above. The fine particle size is especially preferable for cotton in view of the normally short washing times (e.g. 10 minutes), to promote quick dispersability of the compound in the detergent wash solution for maximum effectiveness.

Of course, it is contemplated within the scope of the present invention that the clay-like materials disclosed herein may be used in combination with commercial SARD agents such as CMC and PVP to improve their SARD properties.

The following typical wash test procedure is used to evaluate the effectiveness of the synthetic clay-materials as SARD agents in detergent formulations:

1. Turn the tergotometer heater switch on and adjust the thermostat to 120°F. Set agitator speed at 100 cycles per minute.

2. Weigh out one 1.50 gram of each detergent formulation sample (which may contain a SARD agent) to be tested.

3. Add each weighed amount to a tergotometer bucket filled with 975 ml. of tap water. Agitate this mixture 30 seconds.

4. Add 25 ml. of 2% Aquadag (registered Trade Mark) solution (made by mixing into 1000 ml. of deionized water 20 g. of Aquadag stock solution, which contains 22% solids of colloidal graphite in water) to each bucket containing detergent and water. Agitate this for 30 seconds.

- 5—A. Add to each bucket the following standard test fabric swatches identified with laundry ink.

Eight white pieces of 80 × 80 cotton (3" × 6") folded in thirds.

Four white pieces of Dacron/cotton 7406 WRL (3" × 6") folded in thirds.

- 5—B. Or add to each bucket the following 3" × 6" fabric swatches folded in thirds.

| | | |
|------------------------|-----------|-----|
| Dacron/cotton 7406 WRL | —2 pieces | 110 |
| Dacron/cotton 7406 | —1 piece | |
| Cotton 400W | —3 pieces | |
| Dacron spun 754 AW | —1 piece | |
| Nylon spun 358 | —1 piece | |
| Dacron/cotton 7402A | —1 piece | 115 |
| Acetate Jersey S/113 | —1 piece | |
| Spun Viscose S/266 | —1 piece | |
| Orlon—75 S/862 | —1 piece | |

Total 12 pieces 120
("Dacron" and "Orlon" are Registered Trade Marks)

6. Allow tergotomer to agitate 10 minutes.
7. After completion of cycles remove fabrics and agitators. Squeeze excess liquid from swatches.
- 5 8. Add 1000 ml. of 100°F. water to each bucket. Place the swatches in same tergotometer bucket and rinse for 5 minutes at 100 cycles per minute agitation.
9. Remove swatches from the bucket and
- 10 squeeze the excess water out and dry.
10. After the fabric is completely dry and conditioned to room temperature reflectance readings are taken using a standard Reflectometer. Using white tile as the reference
- 15 standard, take the reading through a single thickness of 80 × 80 cotton. To take readings on Dacron/cotton arrange the four swatches from a bucket in sandwich form for reflectance measurements. When comparing detergent
- 20 formulations, lower reflectance readings after washing indicate lower SARD effectiveness, on a given test fabric.
- In the above description of a typical wash test procedure, it should be noted that a tergotometer is a standard instrument for testing
- 25 detergency efficiency, usually consisting of four numbered 1.5 litre pots containing agitators which are used to simulate typical laundering conditions.
- 30 Also, in step 5A or 5B the standard test fabrics used are identified by a number assigned by the supplier of these fabrics—Testfabrics, Inc., New York City—and are as follows:
- 35 1. Dacron/Cotton—style 7406 WRL— 65/35 shirting with permanent press finish.
2. Cotton—style 400 W—Bleached 80 × 80 cotton print cloth.
3. Dacron/Cotton — Styde 7406 — 63/35
- 40 shirting.
4. Acetate Jersey — Style 113 — All de-lustered filament.
5. Spun viscose—Style 266—Challis (print cloth).
- 45 6. Dacron type 54—Style 754 AW—100% spun fabric.
7. Orlon—75—Style 862—Sand Weave.
8. Nylon spun 6.6—Style 358.
9. Dacron/cotton — Style 7402A — 65/35
- 50 poplin (raincoat weight).
10. Dynel — Style 902 — modified acrylic (this fabric sample was not used in all the tests).
- Typically, the clay materials of the invention
- 55 have relatively high surface areas compared to natural clays, being about 100—600 meters²/gram.
- It has been known in the past that natural clays (e.g. Ben A Gel and sodium bentonite
- 60 BH—200) have soil suspending properties, however as shown in Example VIII, their SARD properties are truly inferior to the clay materials of this invention. ("Ben A Gel" is a Registered Trade Mark).
- 65 Effective amounts of SARD agents in com-

mercial detergent formulation are typically 0.5—1.5%, based on the weight of the formulation. Preferably, about 1% by weight of the compounds of the invention are incorporated as SARD agent.

Use of the compounds of the invention in detergent formulations contributes to the anti-static properties of the fabric.

The following examples are provided for illustrative purposes and should not be interpreted as limiting the invention, the scope of which is defined by the appended claims.

Example I

A) A compound (where X is 0.7) having the formula



is prepared by first forming a slurry of the ingredients by co-precipitation as follows:

A stirred solution containing 5310 grams of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ dissolved in 25 liters of water and heated to about 205°F. in a 60 liter tank by means of steam coils, is precipitated over a 29 minute period with an alkaline solution having a temperature of about 140°F. and prepared by dissolving 6810 grams of N sodium silicate (8.6% Na_2O , 28.6% SiO_2) and 1285 grams of Na_2CO_3 in 25 liters of hot water. At the end of the precipitation period the precipitate slurry temperature is about 198°F. This is rapidly increased to and maintained at about 206°F. while the slurry is digested for a period of 1-1/2 hours during which time additional gas, taken to be CO_2 , is evolved from the slurry. The slurry is then filtered and the resulting filter cake washed with 30 liters of water and allowed to drain on the filter. The washed filter cake, having a total weight of about 22,390 grams, is stirred to a viscous fluid into which is stirred a solution containing 350 grams of NaOH in 750 ml. of water. Then this material is placed in pans and subjected to elevated temperature, about 365°F., at the corresponding gauge pressure, about 150 psi, in a steam operated, horizontal autoclave for about 16 hours. The autoclave product cake is cut into small pieces, dried, disintegrated in a hammer mill and then pebble mill ground to a fine powder (to about 5% plus 325 mesh).

B) Alternatively, the compound having the same above chemical formula is prepared by first forming the slurry by pebble milling as follows;

Seven batches are made as follows and blended. 200 ml. of water are stirred into 478 grams of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ crystals. To this is added a solution containing 612 grams of N sodium silicate, 91 grams of NaOH and 300 ml. of water. The batch is stirred to a fluid, gritty mass. The blend is then ground in a vertical, vibrating pebble mill for a period of one hour. The mill is discharged and rinsed

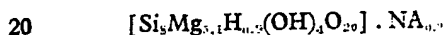
with 6300 ml. of water. The resulting, stirred
slurry is filtered and the filter cake is washed
with 14,000 ml. of water and allowed to drain
on the filter. The washed filter cake, having a
total weight of about 7080 grams is stirred to
a viscous fluid into which is stirred a solution
containing 123 grams of NaOH in 350 ml. of
water. This material is autoclaved as above
and dried. Two batches of dried product are
produced, blended and ground to a fine powder
as above.

Example II

The procedure of Example IA or IB is fol-
lowed to produce a compound having the same
chemical formula but the final product is
ground to smaller than 60 mesh and is not
as fine as 325 mesh.

Example III

A compound having the formula

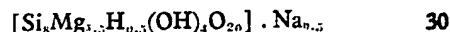


is prepared by following the procedure of
Example IB, except that:

460 grams of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ are used in-
stead of 478 grams; 83.3 grams of NaOH
are used instead of 91 grams; and the final
product is ground to smaller than 60 mesh
rather than 325 mesh.

Example IV

A compound having the formula



is prepared by following the procedure of
Example IB, except that:

495 grams of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ are used
instead of 478 grams; 93.8 grams of NaOH
are used instead of 91 grams; and the final
product is ground to smaller than 60 mesh
rather than 325 mesh.

Example V

The standard wash procedure previously
described is followed in which the following
detergent formulation samples in Table 1 are
tested for SARD properties:

TABLE 1

| | Sample No. | | | | | |
|---|------------|----------|--------|----------|----------|----------|
| | 1 | 2 | 3 | 4 | 5 | 6 |
| Colgate Detergent Base | 1.485 g. | 1.485 g. | | 1.485 g. | 1.485 g. | 1.485 g. |
| C.M.C. | | 0.015 g. | | | | |
| Ajax (Registered Trade Mark) Detergent | | | 1.5 g. | | | |
| P.V.P. K-30 | | | | 0.015 g. | | |
| Compound prepared in Example IA | | | | | 0.015 g. | |
| Compound prepared in Example IB | | | | | | 0.015 g. |

Note in Table 1 that Ajax detergent has the following composition shown in Table 2.

TABLE 2

| | | |
|----|---|----------|
| 45 | Anionic surfactant | 10 wt. % |
| | Nonionic surfactant | 2 |
| | Soap (e.g. sodium or potassium stearate) | 2 |
| | Sodium tripolyphosphate | 35 |
| 50 | Sodium Silicate | 7 |
| | SARD agent (mixture of CMC and PVA) | 1 |
| | Sodium sulfate | 33-35 |
| | Water | 8-10 |

The Colgate detergent base has the same
composition as Ajax, but with no SARD agent.

Also, note that,
PVP/K-30 means polyvinylpyrrolidone
(Avg. mol. wt. 40,000);
CMC means carboxymethylcellulose;
and PVA means polyvinyl alcohol.

The following reflectance readings for each
of the six samples previously mentioned in
Table 1 are given for fabric samples according
to step 5A (Table 3) and step 5B (Table 4)
in the standard wash test procedure.

TABLE 3

| Fabrics | Average Reflectance Before Washing | Reflectance after Washing | | | | | |
|--|--|---------------------------|-------|-------|-------|-------|-------|
| | | 1 | 2 | 3 | 4 | 5 | 6 |
| Dacron/cotton — 7406 WRL 1st Piece | 89.13 | 66.07 | 69.14 | 73.06 | 78.09 | 74.38 | 74.01 |
| Dacron/Cotton — 7406 WRL 2nd Piece | 88.92 | 65.94 | 68.06 | 73.19 | 79.21 | 74.79 | 74.21 |
| Dacron/Cotton — 7406 WRL 3rd Piece | 89.16 | 65.58 | 68.19 | 72.53 | 78.62 | 73.57 | 74.50 |
| Dacron/Cotton — 7406 WRL 4th Piece | 89.02 | 67.47 | 69.27 | 73.64 | 77.75 | 74.58 | 74.94 |
| Cotton — 400 W 1st Piece | 94.08 | 41.37 | 61.34 | 66.28 | 52.80 | 63.56 | 59.20 |
| Cotton — 400 W 2nd Piece | 94.39 | 43.60 | 60.01 | 64.02 | 52.14 | 74.56 | 60.60 |
| Cotton — 400 W 3rd Piece | 94.22 | 44.11 | 61.94 | 64.61 | 52.98 | 63.97 | 60.05 |
| Cotton — 400 W 4th Piece | 94.18 | 45.26 | 61.63 | 65.35 | 52.11 | 64.82 | 61.54 |
| Cotton — 400 W 5th Piece | 94.97 | 43.55 | 61.42 | 65.13 | 52.10 | 64.91 | 60.92 |
| Cotton — 400 W 6th Piece | 95.03 | 43.39 | 61.87 | 65.10 | 52.99 | 64.60 | 61.08 |
| Cotton — 400 W 7th Piece | 95.21 | 43.50 | 60.92 | 64.73 | 51.87 | 63.91 | 61.19 |
| Cotton — 400 W 8th Piece | 94.20 | 43.36 | 60.80 | 65.75 | 52.11 | 64.41 | 60.84 |

TABLE 4

| Fabrics | Average Reflectance Before Washing | Reflectance after Washing | | | | | |
|--|--|---------------------------|-------|-------|-------|-------|-------|
| | | 1 | 2 | 3 | 4 | 5 | 6 |
| Dacron/Cotton — 7406 WRL 1st Piece | 91.69 | 69.19 | 70.51 | 76.22 | 78.09 | 77.21 | 77.94 |
| Dacron/Cotton — 7406 WRL 2nd Piece | 91.18 | 70.52 | 72.03 | 78.10 | 79.21 | 77.37 | 78.21 |
| Dacron/Cotton — 7406 | 92.61 | 41.84 | 44.65 | 52.19 | 62.59 | 72.71 | 69.22 |
| Cotton — 400=W 1st Piece | 94.19 | 46.52 | 64.72 | 66.97 | 53.36 | 64.51 | 60.22 |
| Cotton — 400=W 2nd Piece | 94.30 | 47.08 | 65.45 | 64.49 | 53.53 | 65.77 | 59.92 |
| Cotton — 400 W 3rd Piece | 93.71 | 45.98 | 63.90 | 67.22 | 52.27 | 64.28 | 60.91 |
| Dacron Spun 754 AW | 90.86 | 77.67 | 77.95 | 81.31 | 82.03 | 82.18 | 83.34 |
| Nylon Spun 358 | 89.19 | 73.02 | 75.21 | 77.39 | 82.84 | 78.28 | 82.29 |
| Dacron/Cotton 7402A | 87.43 | 27.99 | 27.05 | 28.28 | 31.07 | 56.41 | 51.81 |
| Acetate Jersey S/113 | 90.11 | 31.34 | 32.25 | 48.32 | 73.65 | 73.37 | 68.80 |
| Spun Viscose S/266 | 91.75 | 68.21 | 73.84 | 78.56 | 77.56 | 80.90 | 82.92 |
| Orlon—75—S/862 | 89.89 | 83.32 | 81.77 | 83.25 | 84.32 | 84.83 | 84.14 |

Example VI

The standard wash. procedure is similarly followed as in Example V, except that step 5. 5B fabrics are only tested and the detergent formulation tested contains 1.485 g. of Colgate

detergent base and 0.015 g. of the compound as prepared in Example III. Reflectance values before and after washing are presented in Table 5 below.

TABLE 5

| <i>Reflectance Before Washing</i> | | <i>Reflectance after Washing</i> | | |
|-----------------------------------|----------------------------------|----------------------------------|----------------------------------|-------|
| 5 | Dacron/Cotton 7406 WRL—1st piece | 92.17 | Dacron/Cotton 7406 WRL—1st piece | 73.05 |
| | Dacron/Cotton 7406 WRL—2nd piece | 91.84 | Dacron/Cotton 7406 WRL—2nd piece | 74.96 |
| | Dacron/Cotton 7406 | 92.42 | Dacron/Cotton—7406 | 44.28 |
| | Cotton 400=W | 95.16 | Cotton—400 W | 46.60 |
| | Cotton 400=W | 95.16 | Cotton—400 W | 43.44 |
| 10 | Cotton 400=W | 95.16 | Cotton—400 W | 43.44 |
| | Dacron 754=W | 89.49 | Dacron 754 W | 62.14 |
| | Nylon 358 | 89.99 | Nylon 358 | 78.26 |
| | Dacron/Cotton 7402 A | 90.37 | Dacron/Cotton—7402 A | 26.69 |
| | Acetate Jersey S113 | 89.37 | Acetate Jersey—S113 | 30.51 |
| | Spun Viscose S266 | 89.80 | Spun Viscose—S266 | 69.85 |
| | Orlon—75—S862 | 87.19 | Orlon—75—S—862 | 83.39 |

15

Example VII

The standard wash procedure is similarly followed as in Example V, except that step 5A fabrics are only tested and the detergent formulation tested contains 1.485 g. of Colgate

detergent base and 0.015 g. of the compound as prepared in Example IV. Reflectance values before and after washing are presented in Table 6 below.

20

TABLE 6

| | Reflectance Before Washing | Reflectance After Washing |
|---------------------------------------|-------------------------------|------------------------------|
| Dacron/Cotton — 7406 WRL 1 piece | 92.11 | 75.33 |
| Dacron/Cotton — 7406 WRL 2nd piece | 91.83 | 77.41 |
| Dacron/Cotton — 7406 WRL 3rd piece | 92.16 | 76.46 |
| Dacron/Cotton — 7406 WRL 4th piece | 90.80 | 76.29 |
| Cotton — 400 W — 1st piece | 93.99 | 55.56 |
| Cotton — 400 W — 2nd piece | 93.64 | 56.35 |
| Cotton — 400 W — 3rd piece | 94.19 | 55.81 |
| Cotton — 400 W — 4th piece | 94.82 | 55.93 |
| Cotton — 400 W — 5th piece | 94.16 | 56.02 |
| Cotton — 400 W — 6th piece | 94.13 | 56.19 |
| Cotton — 400 W — 7th piece | 94.21 | 55.87 |
| Cotton — 400 W — 8th piece | 94.72 | 56.23 |

25

Example VIII

This example compares the SARD effectiveness in detergent formulations of natural clay materials such as Ben A Gel (see data in

Example IX) and Sodium Bentonite BH—200 (see data in Example IX) against the compound as prepared in Example II.

30

The standard wash procedure is similarly

5 followed as in Example V, except that step 5B fabrics are only tested and the detergent formulations tested contain 1.4888 g. of Colgate detergent base and 0.0112 (0.75%) of SARD agent each of 3 samples as follows:

Sample 1: SARD agent is compound of Example II
 Sample 2: SARD agent is Ben A Gel
 Sample 3: SARD agent is Sodium Bentonite BH—200

10

Reflectance values before and after washing are presented in Table 7 below.

TABLE 7
 Reflectance Before Washing

| | 1 | 2 | 3 |
|-------------------------------------|-------|-------|-------|
| Dacron/Cotton 7406 WRL—1 | 90.36 | 90.70 | 91.65 |
| Dacron/Cotton 7406 WRL—2 | 90.87 | 90.91 | 91.85 |
| Dacron/Cotton 7406 | 91.44 | 91.07 | 92.28 |
| Cotton 400 W—1 | 92.97 | 93.90 | 95.54 |
| Cotton 400 W—2 | 93.04 | 93.58 | 94.88 |
| Dacron Spun 754 AW | 89.24 | 89.27 | 89.53 |
| Nylon Spun 358 | 90.58 | 90.73 | 90.28 |
| Dacron/Cotton 7402 A | 91.00 | 91.96 | 91.99 |
| Acetate S/113 | 92.21 | 93.09 | 93.89 |
| Viscose S/266 | 92.25 | 92.26 | 92.18 |
| Orlon—75—S/862 | 86.60 | 88.15 | 87.19 |
| Dynel (Registered Trade Mark) S/902 | 85.23 | 85.93 | 86.60 |

Reflectance After Washing

| | 1 | 2 | 3 |
|--------------------------|-------|-------|-------|
| Dacron/Cotton 7406 WRL—1 | 79.88 | 78.80 | 76.62 |
| Dacron/Cotton 7406 WRL—2 | 80.93 | 76.29 | 76.75 |
| Dacron/Cotton 7406 | 68.71 | 49.84 | 56.58 |
| Cotton 400 W—1 | 55.39 | 45.39 | 48.32 |
| Cotton 400 W—2 | 54.38 | 42.39 | 47.42 |
| Dacron Spun 754 AW | 82.74 | 64.87 | 71.71 |
| Nylon Spun 358 | 79.78 | 74.28 | 73.35 |
| Dacron/Cotton 7402A | 43.04 | 26.05 | 27.96 |
| Acetate S/113 | 48.94 | 22.24 | 26.43 |
| Viscose S/266 | 82.65 | 67.50 | 73.13 |
| Orlon—75—S/862 | 84.34 | 79.94 | 81.08 |
| Dynel S/902 | 80.67 | 77.55 | 79.05 |

EXAMPLE IX

TABLE 8

X-Ray Diffraction and Nitrogen Surface Area Data

| Sample | Crystalline Size Å | Surface Area m ² /g. |
|---------------------------|-----------------------|------------------------------------|
| Compound from Example IA | 56 | 109 |
| Compound from Example IB | 68 | 240 |
| Compound from Example III | <50 | — |
| Compound from Example II | — | 537 |
| *Ben A gel | 200 | 52 |
| *Sodium Bentonite, BH—200 | 238 | 31 |

*Typical Analysis

Note that compounds prepared in Example I and Example II were prepared in a similar manner except that the autoclave product of Example II was washed prior to drying. It is therefore believed that the surface area listed for II is a true figure for the actual clay product and that the surface area for the clay portion of I should be much higher but was masked by the presence of the sodium

hydroxide in these samples which was not washed away.

Crystalline size is expressed as a dimension in terms of angstrom units of length. The crystalline is bounded by a change in electron density and the size is measured in a direction perpendicular to the characteristic 060 crystallographic plane of the (montmorillonite) unit cell.

| | Ben A Gel | Sodium Bentonite, BH—200 |
|-------------------------------------|-----------|--------------------------|
| SiO ₂ | 56.5 | 64.72 |
| Al ₂ O ₃ | 0.2 | 20.82 |
| Fe ₂ O ₃ | 0.2 | 3.44 |
| TiO ₂ | — | 0.14 |
| MgO | 25.8 | 2.38 |
| CaO | 2.8 | 0.49 |
| Na ₂ O, K ₂ O | 2.6 | 2.92 |
| Li ₂ O | 1.1 | — |
| Cl | 2.5 | — |
| F | 1.0 | — |
| Ignition Loss | 7.7 | 4.84 |
| | 100.4 | 99.75 |

Example X

5 The standard wash procedure previously described is followed in which detergent formulation samples containing CMC, the compound prepared by Example IA, and the compound prepared by Example IB are evaluated for SARD effectiveness at varying

concentrations of the above SARD agents—no SARD, 0.25%, 0.50%, 0.75%, and 1.00% (all in weight % based on total weight of 1.5 grams for detergent formulation plus SARD agent). Table 9 shows the results based on reflectance readings before and after washing.

10

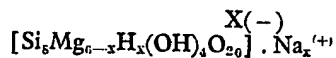
TABLE 9

| Fabrics | Average Reflectance Before Washing | No S.A.R.D. | 0.25% C.M.C. | 0.25% Ex. IA | 0.25% Ex. IB | 0.50% C.M.C. |
|-----------------------------|--|----------------|-----------------|-----------------|-----------------|-----------------|
| 7406 WRL—1 Dacron-Cotton | 91.08 | 68.28 | 66.30 | 69.91 | 70.17 | 66.10 |
| 7406 WRL—2 Dacron-Cotton | 91.22 | 68.03 | 67.07 | 70.68 | 68.62 | 67.87 |
| 7406 WRL Average | 91.15 | 68.15 | 66.69 | 70.29 | 69.39 | 66.99 |
| 7406 Dacron-Cotton | 91.32 | 41.84 | 42.75 | 59.75 | 55.05 | 44.28 |
| 400 W—1 Cotton | 93.90 | 50.81 | 54.81 | 54.28 | 52.48 | 60.25 |
| 400 W—2 Cotton | 94.22 | 48.08 | 56.40 | 56.11 | 50.89 | 60.49 |
| 400 W—3 Cotton | 93.89 | 49.30 | 55.19 | 55.81 | 49.87 | 60.09 |
| 400 W—4 Cotton | 94.13 | 49.61 | 55.55 | 53.87 | 48.59 | 61.02 |
| 400 W Average | 94.03 | 49.45 | 55.49 | 55.02 | 50.46 | 60.46 |
| 754 AW Spun Dacron | 89.78 | 76.82 | 78.74 | 78.73 | 80.08 | 77.61 |
| 358 Spun Nylon | 89.95 | 71.94 | 72.89 | 78.02 | 78.51 | 74.13 |
| 7402 A Dacron-Cotton | 88.19 | 27.33 | 27.35 | 41.24 | 36.92 | 23.19 |
| S/113 Acetate Jersey | 91.11 | 30.43 | 31.23 | 63.75 | 57.70 | 30.88 |
| S/266 Dynel | 84.90 | 63.00 | 69.44 | 76.11 | 73.49 | 70.72 |

| Fabrics | Reflectance After Washing | | | | | | | |
|-----------------------------|---------------------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| | 0.50% Ex. IA | 0.50% Ex. IB | 0.75% C.M.C. | 0.75% Ex. IA | 0.75% Ex. IB | 1.00% C.M.C. | 1.00% Ex. IA | 1.00% Ex. IB |
| 7406 WRL—1 Dacron-Cotton | 71.16 | 71.02 | 67.95 | 72.91 | 72.92 | 68.49 | 72.78 | 73.95 |
| 7406 WRL—2 Dacron-Cotton | 71.02 | 71.31 | 68.03 | 73.05 | 73.05 | 68.90 | 72.48 | 74.01 |
| 7406 WRL Average | 71.09 | 71.16 | 67.99 | 72.98 | 72.99 | 68.70 | 72.63 | 73.68 |
| 7406 Dacron-Cotton | 66.04 | 62.44 | 46.08 | 71.04 | 64.03 | 46.01 | 71.23 | 68.19 |
| 400 W—1 Cotton | 59.59 | 54.43 | 64.91 | 68.15 | 57.17 | 61.26 | 68.48 | 62.40 |
| 400 W—2 Cotton | 59.19 | 54.91 | 65.07 | 68.95 | 57.02 | 68.97 | 68.31 | 63.41 |
| 400 W—3 Cotton | 59.73 | 54.87 | 64.83 | 68.34 | 56.90 | 67.55 | 69.20 | 62.91 |
| 400 W—4 Cotton | 59.22 | 54.30 | 64.79 | 68.20 | 56.31 | 68.01 | 69.02 | 63.02 |
| 400 W Average | 59.43 | 54.65 | 64.90 | 68.41 | 56.85 | 68.19 | 68.75 | 63.68 |
| 754 AW Spun Dacron | 80.72 | 80.81 | 77.93 | 80.12 | 80.38 | 78.12 | 83.13 | 79.59 |
| 358 Spun Nylon | 76.51 | 76.71 | 72.55 | 78.05 | 74.71 | 75.85 | 82.87 | 79.42 |
| 7402 A Dacron-Cotton | 46.81 | 42.67 | 27.92 | 54.15 | 44.74 | 26.16 | 59.17 | 49.76 |
| S/113 Acetate Jersey | 73.33 | 67.72 | 37.82 | 76.56 | 66.29 | 37.40 | 79.90 | 71.83 |
| S/266 Dynel | 77.67 | 78.38 | 73.42 | 76.60 | 80.40 | 75.60 | 74.01 | 82.51 |

WHAT WE CLAIM IS:—

1. A method of washing fabrics with an aqueous detergent solution, whereby the re-deposition of soil upon the fabric from the aqueous solution is substantially reduced, which comprises incorporating in said detergent a compound having the formula:



10 wherein

X is from 0.5 to 0.9

2. The method of claim 1 wherein about

90% by weight of the particles of said compound have a particle size smaller than 325 mesh (U.S. Sieve Standard).

3. The method of claim 2 wherein X is 0.7.

4. The method of claim 3 wherein said fabric comprises cotton.

5. The method of claim 3 wherein said fabric comprises a synthetic fabric.

6. The method of claim 3 wherein said fabric comprises a mixture of cotton and a synthetic fabric.

7. A method according to claim 1 substantially as described.

8. A detergent when used in the method of

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20

25

claim 1 to which there has been imparted soil
anti-redeposition properties substantially as
described.

STEVENS, HEWLETT & PERKINS,
Chartered Patent Agents,
5, Quality Court,
Chancery Lane,
London, W.C.2.
Agents for the Applicants.

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